W.D. #3319B

REQ. NO. 2-9144-2

CONTRACT REPORT NO. OSQ82-00167

RECOMMENDED VALUES FOR REFERENCE URANIUM TAILINGS: RESULTS OF FOUR URANIUM TAILINGS SAMPLE ANALYSIS

Prepared for:
Department of Supply and Services Canada
Room 1-335
Place du Portage, Phase III
11 Laurier Street
Hull, Quebec
K1A 1C9

Prepared by:
Barringer Magenta Limited
3750 - 19th Street N.E.
Suite 105
Calgary, Alberta
T2E 6V2

MICROMEDIA
AUG 29 1983

10 pp

BM-34-82

May, 1983

1. INTRODUCTION

Reliable reference standards are essential for establishing quality control in assay laboratories. Although rock, soil and vegetation standards are readily available from various government agencies it is often difficult to obtain suitable standards for analysis of certain specific materials such as metallic ores or tailings. This report describes the results of analysis of four uranium tailings samples for Ni, Fe, Ti, Al, Ca, Th, U, As, S and SO_4 with the objective of establishing recommended values for reference uranium tailings to be used as statistical means of controlling precision and accuracy in analytical laboratories.

2. ANALYTICAL METHODOLOGY

The following analytical techniques were used to determine Fe, Al, Ca, Ti, Ni, As, Th, U, S and SO_4. Each of the samples submitted was analyzed five times and quality control was monitored by the introduction of the CANMET standards SY-2, SY-3 and MRG-1.

2.1 DETERMINATION OF Fe, Al, Ca, Ti, Ni BY ATOMIC ABSORPTION SPECTROPHOTOMETRY (A.A.)

A 0.25 gram aliquot of each samples was mixed with 2 mls conc. HNO_3 and 2 mls conc. $HClO_4$, refluxed for 3-4 hours in teflon beakers covered by watch glasses. These were then allowed to cool before a further 8 mls of conc. HF were added to the mixture and the contents of the beaker evaporated to dryness. The residue was dissolved in 6 molar HCl and the solution diluted to 25 mls in a volumetric flask. Concentrations of Fe, Al, Ca, Ti and Ni were determined by aspirating the solution into a calibrated IL-457 atomic absorption spectrophotometer.

2.2 DETERMINATION OF ARSENIC BY HYDRIDE ATOMIC ABSORPTION SPECTROPHOTOMETRY

A 0.5 gram aliquot of each sample was fused with potassium bisulphate, leached with 2 mls of 6 normal HCl and the solution made up to 25 mls in a volumetric flask. Arsenic As concentrations were determined by flameless hydride atomic absorption spectrophotometry using a calibrated Varian AA-5 spectrophotometer.

2.3 DETERMINATION OF THORIUM BY NEUTRON ACTIVATION

A 0.5 gram aliquot of each sample was weighed into a teflon vial irradiated for 4-5 days in a Slowpoke reactor and the

concentration of thorium determined by counting.

2.4 DETERMINATION OF URANIUM BY FLUORIMETRY

A 2 ml aliquot of the $HF/HNO_3/HClO_4$ digest solution was extracted into ethyl acetate, the extract fused with a sodium fluoride pellet and the fluorescence measured in the pellet using a calibrated Jarrell-Ash fluorimeter.

2.5 DETERMINATION OF URANIUM BY DELAYED NEUTRON COUNTING

A 1 gram sample was weighed into a plastic vial and irradiated in a small reactor for several hours and the neutron flux of fission products measured to determine uranium concentration in the sample. Values were corrected for the relative abundances of other uranium isotopes in the sample.

2.6 DETERMINATION OF TOTAL SULPHUR

A 0.5 gram aliquot of each sample was weighed into a crucible, mixed with a copper iron conductor and heated by induction in a Leco total carbon-sulphur analyzer. The sample was combusted in an oxygen stream and the SO_2 generated carried into a dilute HCl-KI solution. The iodine generated was back titrated with standard potassium iodate solution using a starch indicator. Concentrations of sulphur were read from a calibrated buret as direct percent sulphur.

2.7 DETERMINATION OF SULPHATE BY ESTIMATION OF SULPHIDE IN THE SAMPLE

A 5 gram aliquot of each sample was mixed with 20 mls of hydriodic acid (50 grams of potassium iodide in 100 mls

distilled water) and 1-2 mls $HgCl_2$ and the mixture warmed in a flask connected to a nitrogen stream to decompose the sulphide minerals. The nitrogen and H_2S gas mixture was passed for 1 hr into a wash bottle containing 25 mls of cadmium acetate (2 grams cadmium acetate in 100 mls distilled water) with 5 mls of normal sodium hydroxide solution. After the gas was passed through the apparatus for 1 hour the contents of the wash bottle was allowed to cool, excess standard iodine solution was added to the solution with suffcient acetic acid to give a final acid concentration of roughly 0.5 normal. The excess was iodine titrated with a standard thiosulphate solution and the concentration of sulphide-sulphur present was estimated. Sulphate was determined by calculating the difference between sulphide sulphur measured with the hydriodic acid reduction and total sulphur determined by Leco combustion technique.

Detection levels for all techniques used to determine elements in the four samples are given in Table 1.

3. RESULTS AND DISCUSSION

Results of analyses of standards UTS 1-4 are given in Table 2 and mean values for fiver determination of each element with relative standard deviations are tabulated in Table 3. With the exception of As in samples UTS 3 and Th in UTS 1 all % RSD values are below 7%. Analytical precision based on repeated determination of SY-2, SY-3 and MRG-1 is better than 4% RSD with the exception of Ni and Sulphide S. The poor precision for Ni may reflect the low element concentration in the samples due to instrumental detection level.

TABLE 1

Detection Levels for Elements Determined by the Different Techniques.

Element	A.A.	FLUO.	NA	DNC	LECO	SULPHIDE
Al_2O_3%	.01					
CaO%	.01					
TiO_2%	.01					
Fe_2O_3%	.01					
Th ppm			1			
U ppm		0.5		0.1		
Ni ppm	5					
S%					.01	.01
SO_4%						.01
As ppm	0.1					

BARRINGER MAGENTA
M8W 5G2
(416) 675 3870
SUITE 105
CALGARY, ALBERTA
T2E 6V2
(403) 276-9701

FILE: 83-0016
DATE: 28/03/83
MATRIX: HF

DEPT. OF ENERGY MINES & RESOURCES,CANMET H. F. SIEGER

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SAMPLE ID	U-FLUO PPM	U-DNC PPM	AL2O3-AA %	CAO-AA %	FE2O3-AA %	TIO2-AA %	NI-AA PPM	AS PPM	S %	TH-NA PPM	SO4= %
UTS-1	55.0	48.9	11.7	7.44	6.78	.97	45	5.2	.95	140	.94
UTS-1	50.0	48.4	11.9	7.44	6.90	.97	45	5.0	.93	140	.93
UTS-1	45.0	50.8	11.9	7.44	6.78	.97	45	5.8	.94	140	.94
UTS-1	50.0	48.6	11.7	7.44	6.90	.97	45	5.2	.93	130	.92
UTS-1	50.0	45.2	11.9	7.44	6.90	.97	45	5.2	.96	130	.95
UTS-2	55.0	56.0	4.98	.66	4.34	.23	25	16.8	3.08	170	3.03
UTS-2	60.0	54.0	4.83	.66	4.47	.23	25	16.8	3.08	170	3.06
UTS-2	55.0	52.3	4.98	.66	4.47	.23	25	16.8	3.13	180	3.09
UTS-2	55.0	55.3	4.98	.66	4.47	.23	25	18.4	3.10	170	3.04
UTS-2	55.0	59.6	4.83	.66	4.47	.23	25	16.8	3.14	170	3.06
UTS-3	550	527	10.8	5.72	4.73	.37	25	3.2	.21	9	.21
UTS-3	500	523	11.0	5.59	4.73	.37	25	4.0	.22	9	.22
UTS-3	500	518	11.0	5.72	4.73	.37	25	3.2	.21	9	.21
UTS-3	550	512	11.0	5.59	4.73	.37	25	4.0	.21	9	.21
UTS-3	500	515	10.8	5.85	4.73	.37	25	3.2	.21	9	.21
UTS-4	950	991	11.9	2.58	3.96	.37	85	32.8	1.73	15	1.73
UTS-4	1000	989	11.7	2.58	3.84	.37	85	32.0	1.69	14	1.69
UTS-4	1050	991	11.7	2.58	3.84	.37	85	33.6	1.69	14	1.68
UTS-4	950	994	11.7	2.58	3.84	.30	85	32.0	1.73	14	1.72
UTS-4	900	989	11.7	2.58	3.84	.30	85	33.6	1.68	14	1.67

TABLE 3

Mean and Percent Relative Standard Deviation Values for Elements Determined in Samples UTS 1-4.

Standard		UTS 1	2	3	4
Al_2O_3	Mean %	11.8	4.92	10.92	1.0
	RSD %	0.93	1.67	1.0	0.76
Fe_2O_3	Mean %	6.85	4.44	4.73	3.86
	RSD %	0.96	1.31	0	1.39
CaO	Mean %	7.44	0.66	5.69	2.58
	RSD %	0	0	1.91	0
TiO_2	Mean %	0.97	0.23	0.37	0.34
	RSD %	0	0	0	1.11
Ni	Mean ppm	45	25	25	85
	RSD %	0	0	0	0
As	Mean ppm	5.3	17.1	3.5	32.8
	RSD %	5.74	4.18	12.44	2.43
Th	Mean ppm	110.2	172	9	14.2
	RSD %	55.5	2.6	0	3.15
U (Flu)	Mean ppm	50	56	520	970
	RSD %	7.07	4.0	5.27	5.88
U (DNC)	Mean	48.4	55.4	519	991
	RSD	4.17	4.9	1.16	0.21
S	Mean	0.942	3.106	0.212	1.704
	RSD	1.38	0.90	2.11	1.41
SO_4	Mean	0.936	3.056	0.212	1.70
	RSD	1.22	0.75	2.10	1.52

Note: An RSD of 0 indicates all measurement element values for the standard are identical to the mean given in the table.

TABLE 4

Analytical Precision (RSD), Mean and Recommended Values for Repeated Analyses of Standards SY-2, SY-3 and MRG-1 Introduced During Analysis of UTS 1-4.

Standard	Fe_2O_3%	Ni ppm	%TiO_2	%CaO	%Al_2O_3	As ppm
SY-2 Mean	6.29	6.5	0.28	7.71	12.15	14
n = 4 RSD%	0	36.6	0	1.00	1.40	3.29
Recommended Value	6.28	10	0.14	7.98	12.12	18
Standard SY-3 Mean	6.47	7.5	0.28	8.20	12.01	15.8
n = 4 RSD%	1.0	38.4	0	8.70	0	2.53
Recommended Value	6.42	11	0.15	8.26	11.80	20.0

Standard	Th ppm	U (Flu) ppm	U (DNC) ppm
SY-2 Mean	380	310	276
n = 4 RSD%	0	3.48	0.4
Recommended Value	380	290	290
SY-3 Mean	1000	731	718
n = 4 RSD%	0	3.27	3.7
Recommended Value	990	650	650

Standard	S%
MRG-1 Mean	0.059
n = 4 RSD	2.40
Recommended Value	0.060

Uncertified GSC Standard (BASL) containing 4.92% S mixed with FeS_2 (100:1 mixture)	S^{2-} (SO_4)
Mean	104 ppm
(n = 2) RSD	10.9%